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Indian Standard

ELECTROLESS NICKEL-PHOSPHORUS COATINGS — SPECIFICATION

UDC 669·248·6

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 9

Metallic and Non-Metallic Sectional Committee, MTD 20

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Metallic and Non-Metallic Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Electroless nickel-phosphorus coatings may be obtained from baths formulated to produce bright, semi-bright or dull deposits. Their appearance depends upon the brightness and smoothness of the substrate. The deposits are normally used without machining or polishing.

These coatings are produced by the catalytic reduction of nickel metal ions in hot, usually mildly acidic aqueous solutions at atmospheric pressure using sodium hypophosphite as the reducing agent. Suitable solutions, proprietary or otherwise, are those that produce a deposit, which will meet the requirements of this specification.

The coatings produced are uniform in thickness on irregularly shaped parts, provided that the processing solution circulates freely over all surfaces.

The coating is a metastable alloy of nickel and phosphorus containing up to 15 percent (m/m) of phosphorus. The physical and chemical properties and the structure of electroless nickel are dependent upon the coating composition, the chemical make-up of the plating bath, the pretreatment of the substrate, and heat treatment after plating.

It should be noted that heat treatments above 220°C may reduce the corrosion resistance of the coatings. Heat treatments up to 200°C used to improve adhesion or to give relief from hydrogen embrittlement do not impair the corrosion resistance, or substantially modify the hardness, or the wear properties of the coatings.

It should be recognized that electroless nickel phosphorus coatings tend to be less ductile than those of electrodeposited nickel Certain coatings of less than 10 µm thickness will generally permit limited deformation without cracking or spalling.

In the preparation this standard assistance has been derived from ISO 4527 : 1987 Autocatalytic nickel-phosphorus coatings — Specification and test methods published by International Organization for Standardization.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ELECTROLESS NICKEL-PHOSPHORUS COATINGS — SPECIFICATION

1 SCOPE

This standard specifies requirements and test methods for electroless nickel-phosphorus coatings.

This standard is not applicable to nickel-boron alloys.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water (*third revision*)
IS 3203 : 1982	Methods of testing local thickness of electroplated coatings (*first revision*)
IS 3554 : 1966	Glossary of terms relating to electroplating
IS 5528 : 1985	Method of testing corrosion resistance of electroplated and anodized aluminium coating by copper accelerated acetic acid salt spray (CASS) test (*first revision*)
IS 6009 : 1970	Method for evaluation of results of accelerated corrosion tests
IS 6910 : 1985	Method of testing corrosion resistance of electroplated and anodized aluminium coatings by acetic acid salt-spray test (*first revision*)
IS 8038 : 1985	Method of testing corrosion resistance of metallic and other non-organic coatings by corrodkote test (*first revision*)
IS 9530 : 1980	Method of testing micro-hardness of electroplated coatings
IS 9844 : 1981	Methods of testing corrosion resistance of electroplated and anodized aluminium coatings by neutral salt spray test

3 DEFINITION

Significant Surface

The part of the article covered or to be covered by the coating and for which the coating is essential for serviceability and/or appearance.

4 INFORMATION TO BE SUPPLIED BY THE PURCHASER TO THE PLATER

4.1 Essential Information

The following information shall be supplied by the purchaser to the plater:

a) Number of this standard;

b) The thickness, in micrometres, of the coating and of any undercoats;

c) Details of significant surfaces, to be indicated on drawings or by the provision of suitably marked samples — similarly, any areas on which electroless nickel coatings may not be present shall be indicated;

d) The sampling procedure to be adopted (*see* **8**);

e) The methods of adhesion testing to be employed (*see* **6.5**); and

f) The nominal composition or specification and metallurgical condition of the basis metal.

4.2 Additional Information

The following additional information may be required and, if so, shall be specified by the purchaser:

a) Any requirements for stress-relieving treatment before electroless plating and/or hydrogen embrittlement reduction after plating (*see* **5.2** and **7.2**);

b) Any requirements for treatment to induce compressive stress (for example peening before plating) (*see* **5.3**);

c) Any special requirements for, or restrictions on, pretreatment;

d) Any special requirements for, or restrictions on, heat treatment (*see* **5.2** and **7**);

e) Any requirements for maximum coating thickness, especially for the build-up of worn or over-machined parts. It shall also be specified whether these thickness shall be as-plated, or as obtained after any machining of the coating;

f) The necessity for degaussing (demagnetizing) steel parts before plating to minimise the inclusion of magnetic particles or swarf into the coating;

g) The final surface roughness of the coating (*see* **6.1** and **6.2**);

h) The hardness of the coating and the method of test to be used in verification (*see* **6.4**, **7** and Annex G);

j) The type, size, extent and location of permissible surface defects in the coating (*see* **6.1**);

k) Any special requirements for the chemical composition of the coating (*see* **6.11**);

m) Any requirements for corrosion resistance;

n) Any requirement in respect of porosity and, where relevent, the method of test (*see* **6.6** and Annex C);

p) Any requirements for wear resistance (*see* **6.9** and Annex J);

q) Any requirements or solderability (*see* **6.10**); and

r) Any other special requirements.

NOTE — Close liaison between designers, manufacturers and platers is desirable in order to obtain satisfactory electroless nickel phosphorus coatings and to avoid adverse effects on the mechanical properties of the article.

5 TREATMENT OF BASIS METAL BEFORE PLATING

5.1 Surface Condition

The significant surfaces shall be examined by the plater using the unaided eye or corrected vision for visible surface defects which may be detrimental to the final finish. Any defects shall be brought to the attention of the purchaser before processing.

5.2 Stress Relief Before Plating

5.2.1 Before being plated, steel parts shall be stress relieved if specified by the purchaser. The appropriate conditions given in Table 1 shall be used, unless otherwise specified.

5.2.2 If stress relief is given after shot peening, the temperature shall not exceed 220°C.

5.2.3 Surface-hardened parts shall be heat treated at 130 to 150°C for not less than 5 h, or for shorter periods at higher temperatures if the resulting loss of surface hardness of the substrate is acceptable.

Table 1 Heat Treatment Conditions for Stress Relief Before Plating

(*Clause* 5.2.1)

Maximum Specified Tensile Strength of Steel MPa		Temperature °C	Time, h (Minimum)
Over	Up to and Including		
—	1 050	Non-required	—
1 050	1 450	190 to 220	1
1 450	1 800	190 to 220[1]	18[1]
1 800	—	190 to 220	24

1) Or for a shorter period at a higher temperature

5.2.4 Stress-relieving heat treatment is not normally required for non-ferrous metals. However, brass and copper alloys that undergo stress or surface cracking in the presence of ammonia or solutions of ammonium salts shall be stress relieved.

5.3 Shot Peening

5.3.1 *General Requirement*

If peening is specified to induce compressive stress, an appropriate method shall be applied as specified by the purchaser [*see* **4.2** (b)]. In order to prevent different stress conditions at the surface, there shall be 100 percent coverage during peening.

NOTE — Since electroless nickel-phosphorus coatings may cause a serious loss of fatigue strength, the introduction of compressive stresses into the surface of parts by shot peening is generally beneficial for both sustained load and fatigue properties. The loss of fatigue strength is due to delayed crack propagation from the coating to the base metal during cyclic loading. The introduction of compressive stresses into the surface of the part by shot peening shall help to minimize the loss of fatigue strength of parts subsequently requiring electroless nickel-phosphorus plating.

Peening using steel balls may produce inclusions in the surface of the substrate which may adversely affect the corrosion resistance of the coating.

5.3.2 *Requirements for Steels*

If shot peening is necessary to improve the fatigue strength, the peening intensity, unless otherwise specified (*see* Note), shall be such that, when measured by the method described in Annex E, the arc height is at least

- 0·3 mm for steels of tensile strength less than 1 100 MPa
- 0·4 mm for steels of tensile strength 1 100 MPa or greater.

Unless otherwise specified the peening shall be performed so that the area concerned is completely covered, that is, the balll marks completely overlap each other.

NOTE — Lower intensities may be necessary on thin sections to avoid distortion, but may not be fully effective in avoiding loss in fatigue strength.

5.3.3 *Requirements for Non-ferrous Metals*

For non-ferrous metals, the peening intensity shall be specified by the purchaser.

5.4 Undercoats

5.4.1 Undercoats may be necessary on certain basis metals for any of the following reasons:

a) to promote adhesion;
b) to prevent diffusion; and
c) to prevent plating bath contamination.

5.4.2 To prevent diffusion and process contamination, a continuous undercoating of thickness 2 to 5 μm of either electroplated copper or electroplated nickel shall be used. Such undercoats shall be applied on basis metals containing more than trace quantities of antimony, arsenic, bismuth, cadmium, lead, magnesium, tin and zinc, except bronzes and brasses.

5.4.3 To promote adhesion of certain metals, a continuous undercoat of thickness up to 2 μm of either electroplated copper or electroplated nickel shall be used. Such undercoats shall be applied on basis metals containing more than trace quantities of chromium, lead, molybdenum, nickel, tin, titanium or tungsten.

6 REQUIREMENTS FOR COATINGS

6.1 Appearance

If specified by the purchaser, the appearance of the significant surface in the as-plated condition shall be bright, semi-bright, or dull. When examined by the unaided eye or corrected vision, the surface shall be uniform and free from defects such as pits, cracks, blisters, exfoliation, or growths except when permitted [*see* **4.2** (g) and (j)].

NOTE — Defects which are present on the basis metal before plating, including hidden defects, may be reproduced by the coating (*see* **5.1**). In addition, stains and discolorations may result from post-plating heat teatment. It is advisable for the interested parties to agree on the acceptability of such defects.

6.2 Roughness

If the roughness is specified by the purchaser, the method of measurement shall be as agreed mutually.

NOTE — It should be recognized that the surface finish of the coating is not usually superior to that of the substrate before plating.

6.3 Thickness

6.3.1 The finished minimum thickness of the electroless nickel-phosphorus alloy plated on the significant surface and of any undercoat (s), shall be specified by the purchaser [*see* **4.1** (b)] and the method of measurement has been specified in Annex A [Guidance on thicknesses to be used is given in Annex H].

6.3.2 If the purchaser specifies the use of test specimen plated simultaneously, as a measure of the electroless nickel coating thickness, this shall be permitted.

6.4 Hardness

Hardness values, if required, shall be measured after heat treatment, and shall be carried out by the method specified in IS 9530 : 1980. The deposit hardness measured shall be within ± 10 percent of that specified by the purchaser (*see* Annex G and **7.3**).

6.5 Adhesion

The electroless nickel-phosphorus coating shall be adherent to the basis material. Coatings shall be capable of passing one or more of the adhesion tests given in Annex B, as specified by the purchaser [*see* **4.1** (e) and **7.4**] in accordance with the following criteria:

a) When tested using the bend test described in **B-1**, no detachment of the coating shall occur. Cracks in the coating on the tension side of the bend shall not be taken as an indication of inadequate adhesion;

b) When tested using the thermal shock test described in **B-2**, no blistering or peeling of the coating shall occur;

c) When tested using the punch test described in **B-3**, no blistering or flaking of the coating shall occur;

d) When tested using the file test described in **B-5**, no lifting of the coating shall be observed.

6.6 Porosity

When tested by the appropriate method described in Annex C and evaluated in accordance with IS 6009 : 1970, the rating shall normally be not lower than 8, unless otherwise specified. However, in some critical applications requiring a total absence of porosity, the rating shall be not lower than 10.

6.7 Corrosion Protection of the Substrates

When specified, electroless nickel-phosphorus coated parts may be subjected to one of the tests specified in IS 5528 : 1985, IS 6910 : 1985, IS 8038 : 1985 and IS 9844 : 1981. The results of the test employed shall be evaluated in accordance with IS 6009 : 1970 and the acceptance level stated by the purchaser.

6.8 Corrosion Resistance of the Coating

If required, the corrosion resistance of the coating shall be specified by the purchaser, together with the method of testing and evaluation.

6.9 Wear Resistance

If required, the wear resistance of the coating shall be specified by the purchaser, together with the method of testing and evaluation [*see* **4.2** (p)]. Annex J gives guidance on the different test methods that are available.

6.10 Solderability

If required, the solderability of the coating shall be specified by the purchaser, together with the method of testing and evaluation.

6.11 Chemical Composition

If required, the chemical composition of the nickel-phosphorus alloy shall be specified by the purchaser [*see* **4.2** (k)] and Annex D]. If the chemical composition is not specified, coatings shall be supplied according to Table 2 and shall conform to the range specified as typical. In this case, verification of chemical composition shall not normally be supplied.

7 HEAT TREATMENT AFTER PLATING

7.1 General

When required by the purchaser, heat treatment after coating shall be carried out:

a) to give hydrogen embrittlement relief (*see* **7.2**),

b) to increase the hardness of the coatings (*see* **7.3**), and

c) to increase the adhesion of the coatings on certain substrates (*see* **7.4**).

If heat treatment for hardening of the coating is carried out, separate hydrogen embrittlement reduction heat treatments may not be needed (*see* **7.2**). Heat treatment shall be carried out befor any mechanical finishing. Delay in applying heat treatment is particularly undesirable with respect to steels of tensile strength of 1 400 MPa and greater (*see* Table 3).

7.2 Heat Treatment for Hydrogen Embrittlement Relief after Plating

7.2.1 The heat treatment of plated steel articles shall be in accordane with the requirements given in Table 3.

The timing of heat treatment shall start from when the article reaches the required temperature and shall not include cooling time.

Table 2 Chemical Composition of the Deposit

(*Clause* 6.11)

Element	Composition, Percentage by Mass		
	Minimum	Maximum	Typical
Nickel	85	98	88 to 95
Phosphorus	2	15	5 to 12
Other elements (Al, As, B, Bi, C, Cd Co, Cr, Cu, Fe, H, Mn, Mo, N, Nb, Pb, S, Sb, Se, Si, Sn, V, Zn)1)	0	2	0·05

1) For certain specific applications, control of these elements within the deposit may be required. Close communications with engineering, quality control and purchasing departments shall help with problems not covered by the essential information when controlling these elements.

7.2.2 Unpeened parts may be heated for a shorter period at a higher temperature if such conditions have been shown to be effective in reducing hydrogen embrittlement. However, hardening of the deposit shall also occur as a consequence (*see* Annex G).

Parts shall not be heat treated to a temperature higher than 50°C below their tempering temperature.

7.2.3 Surface-hardened parts shall be heat treated at 190 to 220°C for not less than 1 h, or at a higher temperature if the resulting loss of surface hardness of the substrate is acceptable.

7.2.4 The specified parts shall be tested after heat treatment by one of the methods described in Annex E.

7.3 Heat Treatment to Increase Hardness

The application of heat-treatment to increase the hardness of electroless nickel-phosphorus coatings has been given in Table 4.

Table 3 Heat Treatment Conditions for Hydrogen Embrittlement Relief After Plating

(*Clause* 7.1)

Maximum Specified Tensile Strength of Steel MPa		Temperature °C	Time h	Maximum Permissible Delay After Plating, h
Over	Up to and Including			
—	1 050	Non-required	—	—
1 050	1 450	190 to 220	8	8
1 450	1 800	190 to 220	18	4
1 800	—	190 to 220	24	0

Table 4 Application of Heat Treatment to Increase Hardness of Autocatalytic Nickel Coatings

(*Clause* 7.3)

Hardness Range (HV)	Nickel-Phosphorus
500 minimum	As deposited
600 to 800	Heat treatment as in Annex G
800 to 1 100 1)	Heat treatment as in Annex G

1) Coatings in this hardness range may be micro-cracked

7.4 Heat Treatment to Improve Adhesion

Heat treatment to improve the adhesion of electroless nickel coatings on certain basis metals shall be carried out as specified by the purchaser or by following the recommendations given in Table 5 for coatings of thickness 50 μm or less on alloys not affected by the temperature given. (Thicker coatings may require proportionately longer times.)

8 SAMPLING

8.1 The sampling procedure shall be as mutually agreed by the purchaser and the plater till Indian Standard is published on sampling.

8.2 When the sampling plan requires the plating of separate test specimens, these shall be plated simultaneously with the articles.

NOTE — The electroless nickel process is subject to rapid changes in solution concentration and daily sampling of the coating deposited is recommended. For coatings specified to have a certain phosphorus content and those requiring corrosion testing, more frequent sampling should be considered as an option [*see* **4.2** (j) and **4.2** (k)].

8.2.1 All such specimens used in the sampling plan shall be made of the same basis material as articles being plated to this specification.

8.3 All specimens shall be provided by the purchaser unless otheirwise agreed by the plater.

Table 5 Recommended Heat Treatment to Improve Adhesion

(*Clause* 7.4)

Material	Time h	Temperature °C
Beryllium and beryllium alloys	1 to 1·5 4	155 ± 5 140 ± 5
Age-hardened aluminium and aluminium alloys	1 to 1·5	130 ± 10
Non age-hardened aluminium and aluminium alloys	1 to 1·5	160 ± 10
Magnesium and magnesium alloys	2 to 2·5	190 ± 10
Copper and copper alloys	1 to 1·5	190 ± 10
Nickel and nickel alloys	1 to 1·5	230 ± 10
Titanium and titanium alloys	10	280 ± 10
Carbon and alloy steels	1 to 1·5	210 ± 10
Molybdenum and molybdenum alloys	2 to 2·5	200 ± 10

9 MARKING

The material may be marked with the Standard Mark.

ANNEX A

(*Clause* 6.3)

DETERMINATION OF COATING THICKNESS

A-1 GENERAL

The thickness shall be measured at any place on the significant surface designated by the purchaser and the measurement shall be made with a measurement uncertainty of less than 10 percent by a method selected by the purchaser. If there is a dispute regarding the thickness, the referee method shall be that specified in **A-3**.

A-2 'WEIGH, PLATE, WEIGH' METHOD

A-2.1 Principle

Plating of a test specimen of similar substrate material to the article being plated. Determination of the increase in mass due to plating. Calculation of the thickness of the plating.

A-2.2 Procedure

A-2.2.1 *Test Specimen*

Determine the area of the test specimen and ensure that it consists of a substrate material similar to that of the article being plated.

A-2.2.2 *Determination*

Weigh, to the nearest 0·001g. Clean and dry test specimen. Plate it under process conditions. Clean and dry the plated test specimen and weigh it to a similar accuracy, at the same temperature used for weighing the uncoated article.

A-2.3 Expression of Results

The coating thickness, in millimetres, is given by the formula:

$$\frac{10\,(m_2 - m_1)}{QA}$$

where

m_1 = mass, in grams, of the test specimen before plating;

m_2 = mass, in grams, of the plated test specimen;

Q = density, in grams per cubic centimetre, of the coating (*see* Table 6);

A = area, in square centimetres, of the plated surface of the test specimen.

NOTE — The density of the coating varies with its phosphorus content. Typical values are given in Table 6.

Table 6 Density of Typical Nickel-Phosphorus Coatings

(*Clause* A-2.3)

Phosphorus Content % (m/m)	Density g/cm^3
6·0	8·0
9·0	7·85
12·5	7·68

A-3 METALLOGRAPHIC SECTIONING

The method specified in IS 3203 : 1982 may be used.

A-4 DIRECT DIMENSIONAL MEASUREMENTS BEFORE AND AFTER PLATING

A-4.1 Principle

Measure the thickness at a specific position on a part or test specimen before and after plating and calculate the thickness of the coating.

A-4.2 Procedure

A-4.2.1 *Test Specimen*

The test specimen shall be either a part or a test piece of similar substrate material to the part.

A-4.2.2 *Determination*

Using a suitable measuring device, for example a micrometer measure, to the nearest 0·002 mm, the thickness of the test specimen at a specific position. Plate it under process conditions. Clean and dry the plated test specimen and measure its thickness, at the same position and to a similar accuracy, at the same temperature used for measurement of the uncoated article.

A-4.2.3 *Expression of Results*

The coating thickness, in millimetres, is given by the formula $\delta_2 - \delta_1$

where

δ_1 = thickness in millimetres, of the test specimen before plating; and

δ_2 = thickness, in millimetres, of the plated test specimen.

In case of test specimens plated on both sides, divide the result obtained from the above formula by a factor of 2.

A-5 COULOMETRIC METHOD

A-5.1 The method specified in IS 3203 : 1982 may be used. The solution recommended by the instrument manufacture, should be used.

NOTE — A 600 g/l solution of sodium nitrate in distilled or deionized water has been found to be suitable for use with some instruments. It is nevertheless essential to check its suitability before using it with a particular instrument.

A-5.2 The instrument shall be calibrated against the coating to be used as the result is significantly influenced by a change in phosphorus content or heat treatment.

NOTE — This method is only recommended for use on steel substrates having a maximum coating thickness of $10\ \mu m$ in the 'as deposited' condition.

A-6 MAGNETIC METHOD

The method specified in IS 3203 : 1982 is applicable to magnetic substrates plated with electroless nickel deposits which contain more than 9 percent (m/m) phosphorus (nonmagnetic) and which have not been heat treated. The instrument shall be calibrated with deposits plated in the same bath, on steel, and whose thickness has been determined by microscopic method described in IS 3203 : 1982.

A-7 SCANNING ELECTRON MICROSCOPE METHOD

A-7.1 Applicability

This clause specifies a method for the determination of metallic coating thickness by examination of cross-sections with a scanning electron microscrope (SEM). It is destructive and has an uncertainty of less than 10% or 0·1 m, whichever is the greater.

A-7.2 Definitions

For the purpose of this clause, the definitions of IS 3554 : 1966 shall apply.

A-7.3 Principal

Cutting, grinding and polishing of a test specimen for metallographic examination by a SEM of a cross-section of the coating.

The measurement is made on a coventional micrograph or a photograph of the video waveform single scan across the coating.

A-7.4 Apparatus

A-7.4.1 *Scanning Electron Microscope (SEM)* having a resolution capability of 50 nm. Suitable instruments are available commercially.

A-7.4.2 *SEM Stage Micrometer* for calibration of the magnification of the SEM. The stage micrometer shall have measurement uncertainty of 5 percent or better for magnification of X 1 000 to X 20 000. Suitable stage micrometers are available commercially.

A-7.5 Procedure

A-7.5.1 *Factors Influencing the Measurement Results*

The following factors may effect the accuracy of a measurement of coating thickness.

A-7.5.1.1 *Surface roughness*

If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces bounding the coating cross-section may be too irregular to permit accurate measurement of the average thickness in the field view.

A-7.5.1.2 *Taper of cross-section*

If the plane of the cross-section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10° to the perpendicular will contribute a 1·5 percent error.

A-7.5.1.3 *Specimen tilt*

Any tilt of the specimen (plane of cross-section) with respect to the SEM beam may result in an inaccurate measurement. The instrument should always be set for zero tilt.

A-7.5.1.4 *Deformation of coating*

Detrimental deformation of the coating can be caused by excessive temperature or pressure during the mounting and preparation of cross sections of soft coatings or coatings melting at low temperatures, and by excessive abrasions of brittle materials during the preparation of cross-sections.

A-7.5.1.5 *Rounding of edges of coating*

If the edge of the coating cross-section is rounded, i.e. if the coating cross-section is not completely flat up to its edges, the observed thickness may differ from the true thickness. Edge rounding can be caused by faulty mounting, grinding, polishing, or etching.

A-7.5.1.6 *Overplating*

Overplating of the test specimen serves to protect the coating edges during preparation of cross-sections and thus to prevent an inaccurate measurement. Removal of the coating material during surface preparation for overplating can cause a low thickness measurement.

A-7.5.1.7 *Etching*

Optimum etching will produce a clearly defined and narrow dark line at the interface of the two metals. A wide or poorly defined line can result in an inaccurate measurement.

A-7.5.1.8 *Smearing*

Polishing may leave smeared metal that obscures the true boundary between two metals and results in an inaccurate measurement. This may occur with soft metals like lead, indium, and gold. To help identify whether or not there is smearing, repeat the polishing, etching, and measurement several times. Any significant variations in readings indicates possible smearing.

A-7.5.1.9 *Poor contrast*

The visual contrast between metals in a SEM is poor when their atomic numbers are close together. For example, bright and semi-bright nickel layers may not be distinguishable unless their common boundary can be brought out sufficiently by appropriate etching and SEM techniques. For some metal combinations, energy dispersive X-ray techniques may be helpful.

A-7.5.1.10 *Magnification*

For a given coating thickness, measurement errors tend to increase with decreasing magnification. If practical, the magnification should be chosen so that the field of view is between 1·5 and 5 times the coating thickness.

The magnification readout of a SEM is often poorer than the 5 percent accuracy often quoted and the magnification has been found for some instruments to vary by 25 percent across the field.

Magnification errors are minimized by appropriate use of a SEM stage micrometer.

A-7.5.1.11 *Uniformity of magnification*

Because the magnification may not be uniform over the entire field, errors can occur if both the calibration and the measurement are not made over the same portion of the field. This can be very important.

A-7.5.1.12 *Stability of magnification*

A-7.5.1.12.1 The magnification of a SEM often changes or drifts with time. This effect is minimized by mounting the stage micrometer and test specimen side by side on the SEM stage so as to keep the transfer time short.

A-7.5.1.12.2 A change in magnification can occur when adjustments are made with the focusing and other SEM electronic controls. Such a change is prevented by not using the focus controls or other SEM electronic controls after photographing the stage micrometer scale except to focus with the x, y and z controls of the stage. Appropriate manipulation of x, y and z controls will bring the specimen surface to the focal point of the SEM beam.

A-7.5.1.13 *Stability of micrographs*

Dimensional changes of micrographs can take place with time, and with temperature and humidity changes. If the calibration micrograph of the stage micrometer scale and the micrograph of the test specimen are kept together and time as allowed for stabilization of the photographic paper, errors from this source will be minimized.

A-7.5.2 *Preparation of Cross-Sections*

Prepare, mount, grind, polish and etch the test specimen so that:

a) the cross-section is perpendicular to the plane of the coating;

b) the surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for the measurement;

c) all material deformed by cutting or cross-sectioning is removed;

d) the boundaries of the coating cross-section are sharply defined by no more than contrasting appearance, or by a narrow, well defined line;

e) if the video waveform signal is to be measured, the signal trace is flat except across the two boundaries of the coating.

A-7.5.3 *Calibration of Instruments*

A-7.5.3.1 *General*

Before use, the SEM shall be calibrated with the SEM stage micrometer (**A-7.4.2**). Appropriate attention shall be given to the factors listed in **A-7.5.1**, to the procedures specified in **A-7.5.4** and to the uncertainty limits of A-7.6 The stability of the calibration shall be checked at frequent intervals.

A-7.5.3.2 *Photography*

Photograph the image of the micrometer scale using a minimum signal to noise ratio of 2 to 1 and with sufficient image contrast for later measurement.

A-7.5.3.3 *Measurement*

A-7.5.3.3.1 Measure the perpendicular centre-to-centre distance between the lines in the photographed image to the nearest 0 01 mm. Use a diffraction plate reader or equivalent device for this measurement.

A-7.5.3.3.2 Repeat the measurement at three different locations at least 3 mm apart on the photograph to determine the average spacing.

A-7.5.3.4 *Calculation of magnification*

The magnification, M, of the photograph is given by the formula:

$$\frac{l_1}{l_2}$$

where

l_1 is the measured distance, in millimetres, between selected lines on the photograph (average of three measurements); and

l_2 is the certified distance, in millimetres, between the lines.

A-7.5.4 *Determination*

A-7.5.4.1 Each instrument shall be operated in accordance with the manufacturer's instructions. Appropriate attention shall be given to the factors listed in **A-7.5.1** and to the uncertainty requirements of **A-7.6**.

A-7.5.4.2 Make a micrograph of the test specimen under the same conditions and instrument settings as used for the calibration and make an appropriate measurement of the micrograph image. Carry out this step in accordance with **A-7.5.4.2.1** or **A-7.5.4.2.2**.

A-7.5.4.2.1 *Conventional micrograph*

With the boundaries of the coatings clearly and sharply defined, make coventional micrographs of the SEM stage micrometer scale and of the test specimen.

Measure the micrographs to at least the nearest 0·1 mm using a diffraction plate reader or equivalent device. If this is not practicable, the sample preparation may not have been suitable.

A-7.5.4.2.2 *Video waveform signal*

Photograph the video waveform signal for a signal scan across the coating, cross-section and across the SEM stage micrometer scale.

To measure the coating, measure the horizontal distance between the inflection points of the vertical portions of the scan at the boundaries of the coatings. Make the measurements to the nearest 0·1 mm using a diffraction plate reader or equivalent device.

A-7.6 Measurement Uncertainty

A-7.6.1 The instrument, its calibration and its operation shall be such that the uncertainty of the coating thickness measurements is less than 0·1 m or 10 percent whichever; is the greater.

NOTE — For a thin gold coating, one laboratory has reported measurement uncertainties of 0·039 for the certification of the SEM stage micrometer scale, 0·02 m for the measurement of the calibration micrographs, and 0·02 m for the measurement ; of the video waveform signal scan.

A-7.7 Expression of Results

The coating thinkness, in micrometre, is given by the formula:

$$\frac{l \times 1\,000}{M}$$

where

l = linear distance, in millimetres, on the micrograph; and

M = magnification (*see* **7.5.3.4**).

A-7.8 Test Report

The test report shall contain the following information:

a) the identification of this test method;

b) the measured value;

c) identication of the test specimen (s);

d) location of measurement on test specimen(s);

e) the magnification, as measured before and after the test specimen measurements;

f) any unusual features of the measurement that might affect the results;

g) date measurements were made;

h) name of the individual responsible for the measurements; and

j) type of measurement : conventional micrograph or video waveform signal.

A-8 PREPARATION OF TEST SPECIMENS AND USE OF SCANNING ELECTRON MICROSCOPE (SEM)

A-8.0 Introduction

The preparation of test specimens and measurements of coating thickness greatly depends on individual techniques and there is a variety of suitable techniques available. It is not reasonable to specify only one set of techniques. The techniques described in this clause are intended for guidance.

A-8.1 Mounting

To prevent rounding of the edge of the coating cross-section, the free surface of the coating should be supported so that there is no space between the coating and its support. This is usually achieved by overplating the coating with at least 10 μm of a metal of hardness similar to that of the coating. The overplate should also give an electron signal different from that of the coating.

A-8.2 Grinding and Polishing

A-8.2.1 It is essential to keep the cross-section surface of the mount perpendicular to the coating. This is facilitated by incorporating additional pieces of a similar metal in the plastic mounting near the outer edges, by periodically changing the direction of grinding (rotating through 90°) and by keeping, reference marks are inscribed; on the sides of the mounts, any inclination from the horizontal is easily measured. Grind the mounted specimens on suitably abrasive paper, using an acceptable lubricant such as water or white spirit, and apply minimum pressure to avoid bevelling the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true specimen profile and to remove any deformed metal. Subsequently, use grades 240, 320, 500 and 600 without exceeding grinding time of 30 to 40s on each paper; alter the direction of grinding by 90° for each change of paper. Then, polishing successively with 6 to 9, 1, and 0·5 μm diamond on microcloth is often recommended.

A-8.2.2 A convenient way to check for tapering of the cross-section is to mount a small diameter rod wire with the specimen so that the perpendicular cross-section of the rod is parallel to that of the coating. If a taper is present, the cross section of the rod will be elliptical.

A-8.2.3 If the video waveform signal scan technique is used, it is important that the scratches be completely removed and that overpolishing does not selectively remove one of the metals more than the other so that the signal scan is distored. With careful polishing, it is unnecessary to use chemical etches.

A-8.3 Use of SEM

A-8.3.1 If the image of the cross-section, as revealed in a conventional micrograph, is to be measured, and if the boundaries of the coating cross-section are revealed solely by the photographed contrast between the two materials, the apparent width of the coating cross-section can vary depending on the contrast and brightness settings. The variation can be as great as 10 percent without any change in instrument magnification. To minimize the resulting uncertainty, adjust the contrast and brightness so that the image shows surface details of the materials on either side of each boundary.

A-8.3.2 Because the magnification of a SEM can change spontaneously with time and can change as a result of changing other instrument settings, it is advisable to calibrate the instrument immediately before or after measurement of the test specimen. For critical measurements, the average of calibration measurements made before and after measurement of the specimen should be used. This ensures that no change in the magnification has occurred and provides information about the precision of the calibration.

A-8.3.3 If the video waveform trace is measured, the measurement is made of the horizontal distance between the inflection points at the boundaries. The inflection point is halfway between the horizontal traces of the two materials. Because this horizontal distance is independent of the contrast and brightness settings and is precisely defined, some operators prefer to measure the video waveform trace for accurate measurements at greater magnifications.

A-8.3.4 For video waveform trace, select a portion of the polished specimen that yields a flat, smooth signal.

A-9 BETA BACKSCATTER METHOD

The coating thickness can be measured by the use of a betabackscatter device, the use of the beta backscatter method is restricted to basis metals which have an atomic number less than 18 or greater than 40. The actual phosphorus content of the coating shall be taken into consideration; consequently, the measuring device shall be calibrated using standard specimens of the same substrate having the same phosphorus content in the coating as the articles to be tested.

ANNEX B

(*Clause* 6.5)

ADHESION TESTS

B-0 Any one of the following methods may be used for the adhesion test.

B-1 BEND TEST

Bend the test specimen through 180° a mandrel of minimum diameter 12 mm, or four times the thickness of the test specimen, and examine at X 4 magnification.

B-2 THERMAL SHOCK TEST

Heat a plated article for 1 h in an oven in accordance with the requirements of Table 7 for the appropriate basis metal.

Table 7 Test Temperatures for Thermal Shock Test

Basis Metal	Temperature (°C)
Steel	200 ± 10
Zinc	200 ± 10
Copper or copper alloys	200 ± 10
Aluminium or aluminium alloys	200 ± 10

Quench the article in water at room temperature.

NOTE — This test procedure may have an adverse effect on mechanical properties of the article under test. Articles so tested should not, therefore, be used for other tests.

B-3 PUNCH TEST

Make several indentations approximately 5 mm apart in the coating using a spring loaded centre punch on which the point has been ground to a radius of 2 mm.

B-4 FILE TEST

If required by the purchaser, a file test may be applied to the article which is coated. File a non-significant area at an angle of 45° to the coating, such that the base metal/coating interface is exposed.

ANNEX C

(*Clause* 6.6)

MEASUREMENT OF POROSITY

C-1 POROSITY TESTS FOR FERROUS SUBSTRATES

C-1.1 Ferroxyl Test

C-1.1.1 *Principle*

Exposure of coated article to potassium ferricyanide [$K_3Fe(CN)_6$] solution by one of the following method:

a) Immersion in a testing solution; and

b) Contact with filter paper impregnated with the test solution.

Observe for formation of either blue spots on pore sites or blue markings on the filter paper in contact with the pore sites.

Estimation of the number of pores over a given area of the article.

C-2 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be applied for the test.

C-2.1 Reagent

C-2.1.1 *Test Solution*

Dissolve 25 g of potassium ferricyanide, and 15 g of sodium chloride in 1 litre of water and rinse.

C-2.1.2 *Impregnating Solutions*

C-2.1.2.1 Dissolve 50 g of sodium chloride and 50 g white gelating in one litre of water (solution *A*).

C-2.1.2.2 Dissolve 50 g of sodium chloride and 1 g of a nonionic wetting agent in one litre of water (solution *B*).

C-2.1.2.3 Dissolve 10 g of potassium ferricyanide in one litre of water (solution *C*).

C-3 PROCEDURE

C-3.1 Test Specimen

Ensure that the test specimen is a clean coated part. Test it by either of the procedures described in **C-3.1.1** and **C-3.1.2.**

C-3.1.1 *Immersion Test*

Immerse the test specimen for 30 s in the test solution (**C-2.1.1**) maintained at 25°C. Remove the test specimen, rinse it in water and allow it to dry in the test specimen, rinse it in water and allow it to dry in the atmosphere. The presence of pore sites is indicated by blue spots visible to the unaided eye.

C-3.1.2 *Impregnated Filter Paper Test*

Immerse strips of 'wet strength' filter paper in solution *A* (**C-2.1.2.1**) maintained at about 35°C. Remove the papers and allow to dry. Immediately prior to the test soak them in solution *B* (**C-2.1.2.2**) and then press them firmly onto the cleaned test specimen, over the area to be tested, ensuring satisfactory contact. Allow the test assembly to stand for 30 min. If the paper strips become dry during the test moisten them in situ with solution *B.*

Remove the paper strips and immediately immerse them in solution *C* (**C-2.1.2.3**) to produce sharply defined blue markings on the paper wherever the basis metal was exposed by discontinuity in the coating, leading to attack by the sodium chloride and transference of the iron components to the paper. If necessary, the same area may be retested.

C-3.2 Expression of Results

Record the number of pore sites per unit area (on either the test article or the paper strip, as appropriate) and indicate whether it exceeds the number permitted by the specification.

C-4 HOT WATER TEST

C-4.1 Principle

Immersion in boiling, aerated water under agreed conditions. Observe for formation of rust spots over pore sites.

C-4.2 Reagent

Aerated water, prepared by bubbling clean, oil-free compressed air, via a glass diffusion disc, through a reservoir of distilled water for at least 24 h. The *p*H of the aerated water shall be 6·7 ± 0·5.

C-4.3 Procedure

C-4.3.1 *Test Specimen*

Ensure that the test specification is a clean coated part.

C-4.3.2 *Test*

Place the test specimen in a suitable vessel and add the aerated water (**C-1.2.2**) at ambient temperature until the specimen is immersed to 30 ± 5 mm. Heat the vessel at such a rate that the water begins to boil in not less than 15 min and not more than 20 min. Immediately remove the test specimen from the water and allow to dry in the atmosphere. Observe the rust spots at the pore sites.

C-4.4 Expression of Results

Record the number of pore sites per unit area and indicate whether it exceeds the number permitted by the specification.

C-5 SALT SPRAY TESTS

Use as appropriate the tests specified in IS 5528 : 1985, IS 6910 : 1985, IS 8038 : 1985 and IS 9844 : 1981, assessing in accordance with IS 6009 : 1970.

C-6 POROSITY TEST FOR ALUMINIUM SUBSTRATES

C-6.1 Principle

Treatment of the test specimen with sodium hydroxide, sodium alizarin sulfonate and glacial acetic acid under agreed conditions. Observe for formation of red spots over pore sites.

C-6.2 Reagents

C-6.2.1 *Sodium Hydroxide*

Dissolve 100 g of sodium hydroxide in one litre of water.

C-6.2.2 *Sodium Alizarin Sulfonate*

Dissolve 1·5 g of methyl cellulose in 90 ml of boiling water. Allow it to cool and add, with stirring, a solution of 0·1 g of sodium alizarin sulfonate in 5 ml of ethanol.

C-6.2.3 *Acetic Acid, Glacial.*

C-6.3 Test-Specimen

Ensure that the test-specimen is a clean coated part.

C-6.3.1 *Procedure*

Immerse the test specimen in or wipe with the sodium hydroxide solution (**C-6.2.1**), allow to stand for 3 min, rinse with water and apply the sodium alizarin sulfonate solution. After 4 min, apply the acetic acid at ambient temperature until the violet colour disappears. Any red spots remaining indicate the presence of pores.

C-6.3.2 *Expression of Results*

Record the number of pore sites per unit area and indicate whether it exceeds the number permitted by the specification.

C-7 POROSITY TEST FOR COPPER SUBSTRATES

C-7.1 Principle

Treatment of a test specimen with glacial acetic acid and potassium ferricyanide solution under agreed conditions. Observe for formation of brown spots over pore sites.

C-7.2 Reagents

C-7.2.1 *Acetic Acid, Glacial*

C-7.2.2 *Potassium Ferricyanide, Solution*

Dissolve 1 g of potassium ferricyanide and 1·5 g of methyl cellulose in 90 ml of boiling water.

C-7.3 Test Specimen

Ensure that the test specimen is a clean coated part.

C-7.3.1 *Procedure*

Immerse the test specimen in or wipe with the glacial acetic acid (**C-7.2.1**) at ambient temperature and allow to stand for 3 min. Apply the potassium ferricyanide solution and allow to stand for 2 to 3 min. Any brown spots indicate the presence of pores.

C-7.3.2 *Expression of Results*

Record the number of pore sites per unit area and indicate whether it exceeds the number permitted by the specification.

ANNEX D

(*Clause* 6.11)

DETERMINATION OF COATING COMPOSITION (NICKEL AND PHOSPHORUS CONTENTS)

D-0 INTRODUCTION

For typical coatings, nickel and/or phosphorus can be determined analytically to confirm the alloy composition. For a typical coating, as specified by the purchaser, confirmation shall be by determination of both nickel and phosphorus.

D-1 PREPARATION OF TEST SPECIMENS

Electroless nickel-phosphorus foil for analysis is prepared by deposition of 20 to 50 μm of electroless nickel on to aluminium foil masked on one side. The aluminium is then removed by immersion in sodium hydroxide solution (10 percent).

D-2 DETERMINATION OF NICKEL CONTENT

D-2.1 Dimethylglyoxime Gravimetric Method

D-2.2 Principle

Dissolution of a test portion in nitric acid. Addition of citric acid to complex any iron present. Neutralization and precipitation of nickel with dimethylglyoxime. Filtration, drying and weighing of the precipitate.

D-2.3 Quality of Reagents

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be applied for the test.

D-2.3.1 *Reagents*

D-2.3.1.1 *Nitric acid,* solution prepared by mixing 1 part by volume of nitric acid (sp. gr. approximately 1·42 g/ml) and 1 part by volume of water.

D-2.3.1.2 *Dimethylglyoxime,* 1 percent (m/v).

D-2.3.1.3 *Citric acid*

D-2.3.1.4 *Dilute Ammonia Solution,* 1 : 1 (*v/v*).

D-2.4 Apparatus

D-2.4.1 *Sintered Glass Crucible*

D-2.4.2 *Oven,* capable of being maintained at 110 ± 2°C.

D-2.5 Procedure

D-2.5.1 Weigh, to the nearest 0·000 1 g, about 0·1 g of the test specimen (*see* **D-1**) and transfer it to a 500 ml beaker.

D-2.5.2 Dissolve the test specimen in 20 ml of the nitric acid solution. Heat the solution so as to boil off any nitrous fumes. Allow the solution to cool to ambient temperature and dilute it to 150 ml with water. Add approximately 1 g of the citric acid and adjust the *p*H of the solution to between 8 and 9 by addition of the ammonia solution (**D-2.3.1.4**).

Bring the temperature of the solution to 60 to 70°C and, while stirring, add 30 ml of the dimethylglyoxime (**D-2.3.2**) solution. Allow the mixture to stand at 60 to 70°C for 1 h, cool it to below 20°C and filter the precipitate using the clean, dry tared sintered glass crucible (**D-2.4.1**). Wash the precipitate well with water, dry in the oven, maintained at 110 ± 2°C, for 1 h. Allow the crucible and contents to cool to ambient temperature in a desiccator and weigh it to the nearest 0·000 1 g.

NOTE — Cobalt and copper are co-precipitated and, if present, should be allowed for in the calculation (*see* **D-2.6**).

D-2.6 Calculation

$$\text{Nickel, percent by mass} = \frac{20{\cdot}32\ (m_2 - m_1)}{m}$$

where

m = mass, in grams, of the test portion;

m_1 = mass, in grams, of the crucible;

m_2 = mass, in grams, of the crucible and precipitate.

NOTE — Where precise measurement of the nickel content is required by the purchaser, the masses of cobalt and copper should be determined and their total mass subtracted from m_2 in the formula given in **D-2.6**.

D-3 DETERMINATION OF PHOSPHORUS CONTENT

D-3.1 Molecular Absorption Spectrometric Method

D-3.1.1 *Principle*

Dissolution of a test portion in nitric acid. Oxidation with potassium permanganate and dissolution of precipitated manganese dioxide with sodium nitrite. Reaction with a solution of ammonia molybdate and ammonium vanadate. Spectrometric measurement of the absorbance of the complex at a wavelength of about 420 nm.

D-3.2 Reagents

D-3.2.1 *Reagent for Dissolution and Oxidation*

D-3.2.1.1 *Dilute nitric acid*, 40 percent (*V/V*).

D-3.2.1.2 *Sodium nitrite solution* 20 g/l (*m/v*).

D-3.2.1.3 *Potassium permanganate solution*, 7·6 g/l (*m/v*).

D-3.2.2 *Reagents for the Determination*

D-3.2.2.1 *Molybdate-vanadate solution*

Dissolve separately in hot water 20 g of ammonium molybdate and 1 g of ammonium vanadate. Mix the two solutions, add 200 ml of nitric acid and dilute to 1 litre with water. Mix well.

D-3.2.2.2 *Phosphorus, standard solution* (1 ml = 0·1 mg P).

Weigh 0·439 2 g of potassium dihydrogen orthophosphate (KH_2PO_4), dissolve it in water and transfer the solution quantitatively to a 1 000 one-mark volumetric flask. Dilute to the mark with water and mix well.

D-3.3 Apparatus

Ordinary laboratory apparatus and spectrophotometer or photoelectric absorptiometer fitted with a filter providing maximum transmission at a wavelength of about 420 nm, equipped with optical cells of path length 10 mm.

D-3.4 Procedure

D-3.4.1 *Preparation of Test Solution*

Weigh, to the nearest 0·000 1 g, 0·19 ti 0·21 g of the the test specimen. Transfer it to a beaker and dissolve in 50 ml of the nitric acid solution. Heat gently until the test portion is fully dissolved. Then boil to remove brown fumes. Dilute the solution to approximately 1 000 ml, bring it to the boil and add 25 ml of the potassium permanganate solution. Boil the solution for 5 min. Add the sodium nitrite solution drop by drop until the precipitated manganese dioxide is dissolved.

Boil the solution for 5 min and then allow it to ambient temperature. Transfer the solution to a 250 ml volumetric flask, dilute to the mark with water and mix well.

D-3.4.2 *Blank Test*

Carry out a blank test in parallel with the determination, by the same procedure, using the same quantities of all the reagents as in the determination, but omitting the test portion.

D-3.4.3 *Preparation of the Calibration Curve*

D-3.4.3.1 Into a series of 100 ml one-mark volumetric flasks, introduce volumes of the phosphorus standard solution shown in Table 8.

Treat the contents of each flask as follows:

Add 25 ml of the molybdate-vanadate reagent, make up to the mark with water and mix. Allow the solution to stand for 5 min. Fill one of the optical cells (**D-3.3**) with the solution.

Table 8 Preparation of Phosphorus Calibration Solutions

(*Clause* D-3.4.3.1)

Volume of Phosphorus Standard Solution ml	Corresponding Mass of Phosphorus P mg
0[1])	0
2	0·2
4	0·4
6	0·6
8	0·8
10	1·0

[1]) Calibration compensation solution.

D-3.4.3.2 Carry out the spectrometric measurements using either the spectrophotometer at the wavelength of maximum absorption (about 420 nm) or the photoelectric absorptiometer fitted with suitable filters after having, in each case, adjusted the apparatus to zero absorption against water. Deduct the absorbance of the calibration compensation solution from those of the other calibration solutions.

D-3.4.3.3 Plot a calibration curve for the masses in mg of phosphorus versus absorbance.

D-3.5 Determination

Transfer 10 ml of the test solution (**D-3.4.1**) to a 100 ml volumetric flask, add 50 ml of water, 25 ml of the molybdate-vanadate solution, make up to the mark with water and mix well. Allow the solution to stand for 5 min. Fill one of the optical cells (**D-3.3**) with the solution.

Carry out the spectrometric measurements as described in **D-3.4.3.2**.

D-3.6 Expression of Results

Use the calibration curve, determine the mass in mg of phosphorus corresponding to the spectrometric measurement.

The phosphorus (P) content, expressed as a percentage by mass, is given by the formula

$$\frac{2{\cdot}5\,(m_3 - m_4)}{m}$$

where

m = mass, in grams, of the test portion;

m_3 = mass, in milligrams, of phosphorus found in the aliquot portion of the test solution used for the determination; and

m_4 = mass, in milligrams, of phosphorus found in the correspoding aliquot portion of the blank test solution.

D-4 DETERMINATION OF PHOSPHORUS CONTENT

D-4.1 Titrimetric Method

D-4.2 Principle

Preparation of the test solution as specified in **D-3.4.1**. Formulation of ammonium molybdophosphate by treatment with ammonium molybdate. Conversion to triammonium orthophosphate; by treatment with potassium nitrate solution. Addition of an excess of standard sodium hydroxide solution and titration of the unreacted sodium hydroxide with volumetric hydrochloric acid.

D-4.3 Reagents for Dissolution and Oxidation

D-4.3.1 *Ammonium Molybdate Solutions*

D-4.3.1.1 *Solution A*

Disssolve 15 g of ammonium molybdate in 80 ml of water. Add 6 ml of ammonia solution (approximately 0·88 g/ml) dilute to 100 ml and mix.

D-4.3.1.2 *Solution B*

Dissolve 24 g of ammonium citrate in 60 ml of water. Add 33 ml of nitric acid, dilute to 100 ml and mix.

D-4.3.1.3 *Preparation*

Mix equal volumes of solutions *A* and *B* while stirring, allow to stand for 12 h and filter if necessary.

D-4.3.2 *Potassium Nitrate Solution*, 10 g/l (*m/v*).

D-4.3.3 *Sodium Hydroxide*, 1·100 mol/l.

D-4.3.4 *Hydrochloric Acid, Solution*, 0·100 mol/l.

D-4.3.5 *Phenolphthalein*, 10 g/l solution in 95 percent (*v/v*) ethanol.

D-4.4 Procedure

D-4.4.1 *Preparation of the Test Solution* (*see* **D-4.3.1**.)

D-4.4.2 Transfer 10 ml of the test solution (**D-4.4.1**) to a stoppered flask and dilute to 100 ml with water.

Warm the solution to 40 to 45°C (do not exceed this temperature) and add 50 ml of the ammonium molybdate reagent (**D-4.3.1**) slowly, while stirring. Stopper the flask. Agitate the flask vigorously for 10 min. Allow the flask to stand for 30 min and filter the reaction mixture.

D-4.4.3 Wash the flask and the precipitate with the potassium nitrate solution until the filtrate will not decolorize 1 ml of water containing 1 drop of the sodium hydroxide solution and 1 drop of the phenolphtalein solution.

Place the filter paper with the precipitate in the original flask, add 50 ml of water, and shake well. Add 10 ml of the sodium hydroxide solution and shake well to dissolve the precipitate. Add the phenolphthalein solution and titrate with the hydrochloric acid solution until the pink colour just appears.

NOTE — About 100 ml of the washing liquid is normally required.

D-4.5 Calculation

The phosphorus (P) content, expressed as a percentage by mass, is given by the formula

$$\frac{25 \times (10 - V) \times 0{\cdot}013\ 49}{m}$$

where

m = mass, in grams, of the test portion; and

V = volume, in millilitres, of the hydrochloric acid standard volumetric solution used in the determination.

D-5 SPECTRAL ANALYSIS

A suitable method using either emission or absorption spectra produced inductively coupled plasma (I C P) would be acceptable for analysis of nickel, phosphorus and other elements.

The following lines have been found to have low interferences when using argon ICP techniques :

Ni 216·86 P 214·94

NOTE — Fresh standards of phosphorus should be used for analysis.

ANNEX E

(*Clause* 7.2.4)

STATIC LOAD TESTING METHOD FOR MEASUREMENT OF EMBRITTLEMENT REDUCTION

E-1 SAMPLE

A radom sample of four parts shall be selected from each inspection lot. Four separately coated specimens may, however, be prepared to represent each inspection lot.

E-2 PROCEDURE

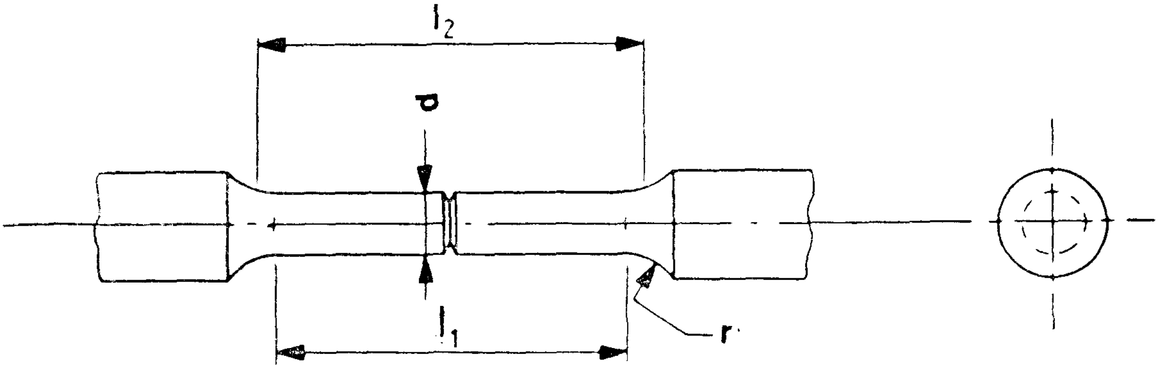
E-2.1 The load, in newtons, or the part deflection or extension, in millimetres, to be used in testing shall be as specified and shall be in accordance with the following:

a) Sample parts such as spring pins and lock rings which are installed in holes or on rods shall be similarly assembled using the applicable parts specifications or drawings tolerances which impose the maximum sustained tensile stress on the coated parts. The parts shall be held under the specified load at 20 ± 1°C for at least 200 h, unless otherwise specified, and then examine for cracks.

b) Sample parts such as pressure containers, fasteners and springs, which in use are subjected to a sustained static tensile load in excess of 25 percent of the basis material specification minimum tensile yield strength, shall be subjected to a sustained load equal to 75 percent of the basis material specification minimum tensile yield strength.

E-2.2 Parts which require special fixtures or extreme loads to comply with these requirements need not be tested as specified, unless in use they are subjected to a sustained static tensile load in excess of 50 percent of the basis material specification minimum tensile yield strength. Parts which require fixture or extreme load to comply with these requirements and which in use are subjected to a sustained static tensile load between 25 percent and 50 percent of the basis material specification minimum tensile yield strength may be represented by separate specimens. Four specimens shall be prepared to represent each lot. Their dimensions shall be in accordance with Fig 1 for rounded specimens.

E-2.3 Specimens shall have a 60° V notch located approximately at the centre of the gauge length. The cross section area at the root of the V shall be approximately equal to half the area of the full cross-section of the specimen's reduced section.



d Diameter	12·5 ± 0·25
l_1 Gauge length	50 ± 0·10
l_2 Length of reduced section, minimum	60
r Radius of fillet, minimum	10

FIG. 1 TEST SPECIMEN, STATICLOAD TEST METHOD FOR MEASUREMENT OF EMBRITTLEMENT REDUCTION

E-2.3.1 The radius of curvature of the *V* at the base of the notch shall be 0·25 ± 0·13 mm. The axis of the round, notched specimen (load direction) shall be perpendicular to the short transverse grain load direction.

E-2.4 These specimens shall be of the same chemical composition (alloy) as the parts represented, shall have been heat treated to the same hardness requirement, and shall have accompanied the production parts, which they represent, through the entire coating process, including all pre-coating and post-coating. The parts of samples shall be held under the specified load at 20 ± 1°C for at least 200 h, unless otherwise specified, and then examined for cracks.

ANNEX F

(*Clause* 5.3.2)

METHOD FOR ESTABLISHING PEENING CONDITIONS

F-1 APPLICABILITY

This annex specifies a method for establishing peening conditions for basis metals, prior to electroplating.

F-2 PRINCIPLE

A rigidly mounted test specimen is peened under specified condition and its curvature, measured after peening, is used as a measure of peening intensity.

F-3 APPARATUS

F-3.1 Test Specimen Mount, as shown in Fig. 2.

F-4 PROCEDURE

F-4.1 Test Specimen

The test specimen comprises a carbon steel sheet, of hardness range 400 HV 30 to 500 HV 30 and of thickness 1·6 mm, which has been cut to a size 75 mm × (20 ± 0·2) mm and ground to a thickness of 1·3 ± 0·02 mm. Ensure that its deviation from flatness does not exceed an arc height of 38 µm when measured as specified in **F-4.2**.

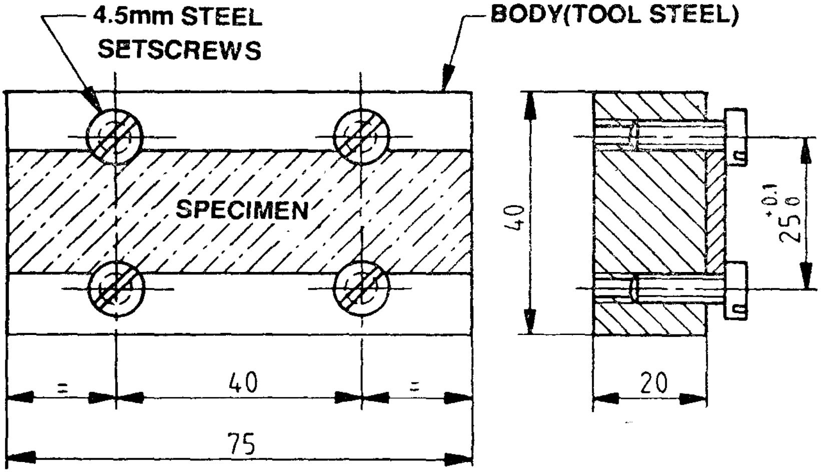
F-4.2 Determination

Fit the test specimen rigidly in its mount (**F-3.1**) and open it on the exposed side for the specified time and conditions.

After peening, remove the test specimen from its mount and measure the curvature of the unpeened surface with a depth gauge, the test specimen being supported on four 5 mm diameter balls, forming a rectangle 32 mm × 16 mm. Align the gauge symmetrically on the test specimen with its centre stylus at the centre of the test specimen. Measure the arc height at the centre of the test specimen over the gauge length of 32 mm, measuring to the nearest 25 µm. Then adjust the peening conditions if necessary, to give the required arc height.

F-5 EXPRESSION OF RESULTS

The peening intensity is expressed as the arc height, in millimetres, after peening, of the unpeened surface of the test specimen.



All dimensions in millimetres.

FIG. 2 TEST SPECIMEN MOUNT

ANNEX G

(*Clauses* 6.4, 7.2.2, and *Table* 4)

HEAT TREATMENT TO INCREASE HARDNESS

G-1 The hardness of the electroless nickel phosphorus coatings may be increased by the following heat-treatment.

G-1.1 Hardness obtained depends upon the temperature, heat treatment time and alloy composition as illustrated by the values in Tables 9 and 10. Graphs of values of hardness after heat treating at various times and temperatures are shown in Fig. 3 and 4. These graphs are correct only for alloy composition within the range 5 percent (m/m) to 10 percent (m/m) phosphorus content. Errors outside this range are particularly marked at temperatures below 350 and above 500°C.

G-1.2 Alternatively, times longer than 1 h at temperature below 400°C can be used to produce the desired effect. Thus Fig. 4 shows that either 400°C for 1 h or 285°C for 12 h will give the maximum hardness.

G-1.3 Rapid heating and cooling should be avoided during heat treatment. The mass of the article should be taken into account when determining heat treatment times. Heat treatment should preferably be carried out in an inert or reducing atmosphere, but a hydrogen atmosphere should not be used for steel parts of minimum specified tensile strength exceeding 1 400 MPa. It should be noted that high heat treatment temperatures may adversely affect the mechanical properties and the corrosion protection of certain basis materials should be taken into account in deciding upon the combination of heat treament time and temperature required to produce the desired final hardness value.

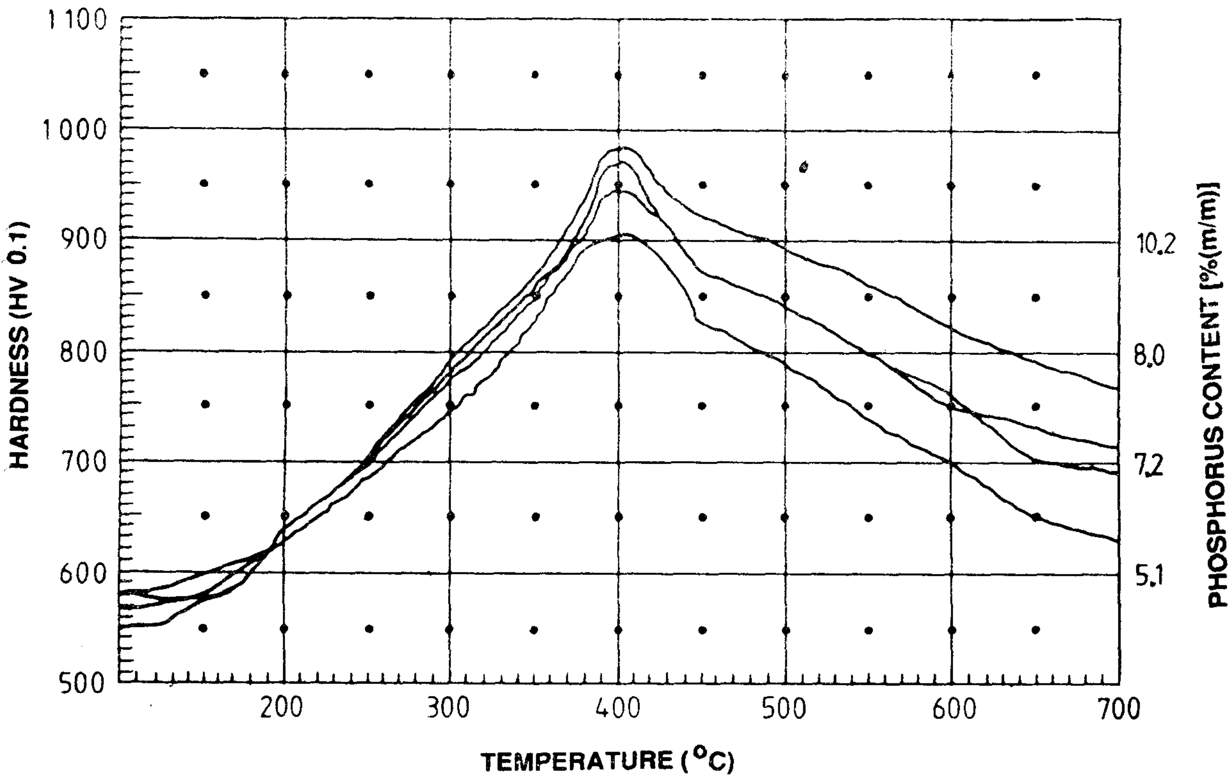


FIG. 3 RELATIONSHIP BETWEEN HARDNESS AND TEMPERATURE

These graphs were produced from the best available information of published sources. Research efforts are under way to re-evaluate the data using ICP and new Hardness Test Equipment. The graphs were produced by heating the test specimen at a rate of 20° C/min to the specified temperature and maintaining it for 1 h.

Table 9 Effect of Duration of Heat Treatment at 400°C on Hardness (H K 0·1)

(*Clause* G-1.1)

Phosphorus Content percent (m/m)	As-plated	After Heat Treatment for 1/4 h	1/2 h	1 h	2 h	20 h
		H K 0.1				
2·8	692	821	—	812	773	—
4·5	732	811	911	923	951	977
6·8	611	782	852	915	957	967
7.1	602	—	921	—	—	916
8·7	584	863	890	893	913	—
9·6	547	—	1 001	—	—	—
12·1	509	845	827	890	766	—
12·5	536	959	961	953	961	960
12·6	568	874	837	845	834	—
12.6	524	862	835	855	831	—

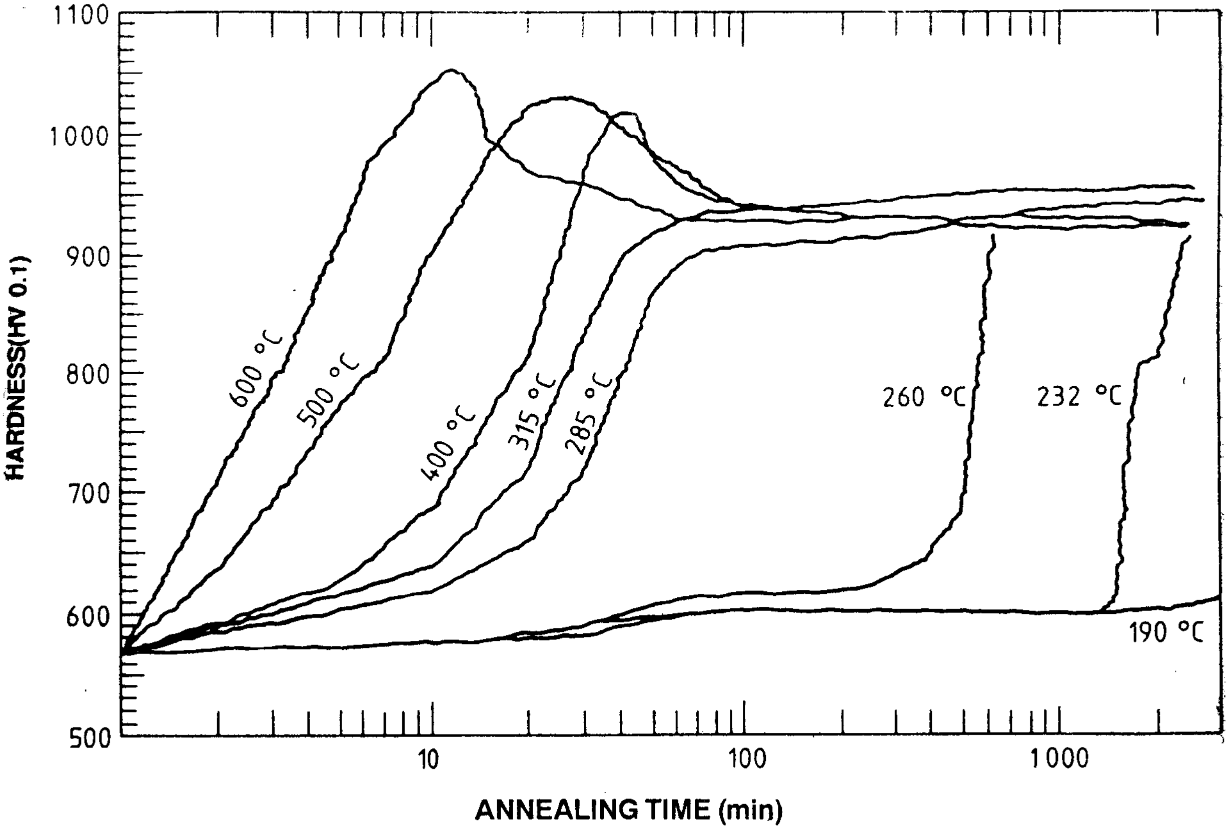


FIG. 4 RELATIONSHIP BETWEEN HARDNESS AND ANNEALING TIME

NOTE — These graphs are average and may vary with deviations from normal practice.

Table 10 Effect of Duration of Heat Treatment Above 400°C on Hardness (H K 0·1)

(*Clause* G-1.1)

Phosphorus Content percent (m/m)	Temperature °C	As-plated	After Heat Treatment for				
			1/4 h	1/2 h	1 h	2 h	20 h
			H K 0·1C				
2·8	600	692	488	423	288	290	221
4·5	425	732			973		793
	500				726		608
	600		539	550	602		
6·8	425	611			1 010		877
	500				926		838
	600		715	717	788	652	575
7·1	425	602			958		765
	500				843		721
12·5	425	536			944		960
	500				903		901
	600		859	846	865	837	731

ANNEX H

(*Clause* 6.3)

GUIDANCE OF THE THICKNESS AND USE OF ELECTROLESS NICKEL PHOSPHORUS

H-0 INTRODUCTION

The principal applications for electroless nickel coatings are described in **H-1** to **H-4.**

H-1 PROVIDING RESISTANCE TO CORROSION

Electroless nickel-phosphorus coatings exhibit excellent corrosion resistance due to their passive nature. If the electroless nickel-phosphorus coating has low porosity, it will afford good corrosion protection to the metal substrate.

The coating thickness required will vary with the corrosion conditions and should be thicker on rough or porous substrates so as to reduce the influence of the substrate surface. To achieve optimum corrosion performance using minimum thickness of coating, the surface condition of the substrate should be as smooth and pore free as is possible.

For mild corrosion protection on smooth, uniform substrates, thickness of 10 to 25 μm for ferrous, non-ferrous and aluminium alloys are recommended. For certain applications, thickness of less than 10 μm have been found to be satisfactory.

For severe corrosion conditions a minimum thickness of 50 μm is recommended.

H-2 PROVIDING RESISTANCE TO WEAR

Thickness of 10 to 25 μm are recommended. For severe wear conditions a thicker coating may be necessary. Heat treatment of the coating will substantially improve the wear resistance of the coating.

H-3 RECLAIMING WORN OR OVER-MACHINED PARTS

Coatings with thickness of 125 to 600 μm may be plated to repair worn parts or to salvage over-machined parts. Undercoats of electrodeposited nickel are sometime applied when total coating thickness exceeds 125 μm. However, machining to size after electroplating may be necessary before application of electroless nickel.

H-4 IMPARTING SOLDERABILITY TO METALS THAT ARE DIFFICULT TO SOLDER (FOR EXAMPLE ALUMINIUM)

The coating thickness is usually 2·5 to 7·5 μm.

A mildly activated rosin flux is required.

ANNEX J

(*Clauses* 4.2 *and* 6.9)

MEASUREMENT OF WEAR RESISTANCE

J-1 TEST METHOD

Wear test methods are specific to different applications. Therefore, only guidelines on the type and method of test to be used have been given. There are a number of suitable wear tests available which include those utilizing the following test principles:

- abrasive wheel;
- oscillating pin producing a notch in the coating;
- block and pin on ring;
- solid particle impingement using a gas jet;
- liquid impingement erosion; and
- vibratory cavitation erosion.

J-1.1 Other suitable test may be agreed upon between the purchaser and the plater, if required.

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